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(पहला पुनरीक्षण)

Method for Determination of Formaldehyde Content in Wood-Based Panels by Extraction Method Called Perforator Method

(First Revision)

ICS 79.060

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Wood and Other Lignocellulosic Products Sectional Committee, CED 20

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Wood and Other Lignocellulosic Products Sectional Committee had been approved by the Civil Engineering Division Council.

This standard was first published in 1993 and covered the method for determination of formaldehyde content in wood-based panels by extraction method called perforator method.

The emission of formaldehyde from a wood-based panel (for example, particleboard, plywood, fibre board and oriented strand board) is a complex process. For a given panel, the test result depends upon the age, ageing conditions, moisture content, etc, at the time of testing.

The perforator value as determined by the method laid down in this standard is considered to be the formaldehyde content of the tested panel. The test result must be considered in relation to the specific board conditions at the time of testing as outlined above. There is not necessarily a correlation between the perforator value and the formaldehyde emission of the wood-based panel.

The temperature of conditioning of wood-based panel has been given as 27 ± 2 °C in **5.3**. However, for the purpose of export of wood-based panels to European countries, values of 20 ± 2 °C may be adopted.

In the formulation of this standard, considerable assistance has been taken from ISO 12460-5 : 2015 ‘Wood-based panels — Determination of formaldehyde release — Part 5: Extraction method (called the perforator method)’

In this revision, the following major modifications have been incorporated:

a) Scope of the standard has been extended to cover testing of all wood-based panels (including wood and other lignocellulosic panel products) in accordance with the standard, and the title of the standard has been accordingly modified, which was earlier limited to only particle board.

b) Limitation of this method for determination of formaldehyde content of 5 mg/100 g and above has been deleted as all the measurement starting from a minimum 0.5 mg/100 g can be determined by titration and spectrophotometer method.

c) Determination of formaldehyde in the extract by the spectrophotometer method has been added as an alternate method for determining the perforator value after the process of extraction in the perforator column.

The Committee responsible for the formulation of this standard is given at Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHOD FOR DETERMINATION OF FORMALDEHYDE CONTENT IN WOOD-BASED PANELS BY EXTRACTION METHOD CALLED PERFORATOR METHOD

(First Revision)

1 SCOPE

This standard covers method for determination of formaldehyde content of unlaminated and uncoated wood-based panels by an extraction method known as perforator method.

NOTE — The reference to wood-based panels mentioned in this standard includes the all wood and other lignocellulosic materials based panel products.

2 PRINCIPLE

2.1 The formaldehyde is extracted from test pieces by boiling in toluene and transferred into distilled or demineralized water. Formaldehyde content in the extract may be determined by any of the two methods, namely titration method and spectrophotometer method.

NOTE — In case of dispute or doubtful marginal values, the titration method shall be taken as referee method.

2.2 In titration method, formaldehyde is oxidized by an excess of iodine in sulphuric acid solution, and unused iodine is back titrated with thiosulphate.

2.3 The spectrophotometer method is based on the Hantzsch reaction in which formaldehyde reacts with ammonium ions and acetylacetone to yield diacetyldihydrolutidine (DDL). DDL has an absorbance maximum at 412 nm. The reaction is highly specific to formaldehyde.

NOTE — Other suitable spectrophotometer procedures may also be used.

3 REAGENTS

For the analysis, following reagents of analytical quality and distilled or demineralized water shall be used.

3.1 Toluene, which is free from water and from impurities which may interfere with the test.

3.2 Iodine Standard Solution, with concentration (I_2) = 0.01 mol/I. Standardization of the solution shall be regularly checked. One millilitre (1 ml) of this solution corresponds to 0.3 mg formaldehyde.

3.3 Sulphuric Acid Solution, obtained by dilution of concentrated sulphuric acid with water at a ratio of 1:1 (volume).

3.4 Sodium Thiosulphate Standard Solution, with concentration ($Na_2S_2O_3$) = 0.01 mol/I.

3.5 Sodium Hydroxide Standard Solution, with concentration (NaOH) = 1 mol/l.

3.6 Starch Solution, 1 percent (m/m).

3.7 Acetylacetone Solution, 4 ml acetylacetone is added to a 1 000 ml volumetric flask, dissolved in distilled water and made up to the mark with distilled water.

3.8 Ammonium Acetate Solution, 200 g ammonium acetate is transferred to a 1 000 ml volumetric flask, dissolved in distilled water, and filled to volume with distilled water.

4 APPARATUS

4.1 Precision Balance, accurate to 0.001 g.

4.2 Well-ventilated Drying Oven, capable of maintaining a temperature of 103 ± 2 °C.

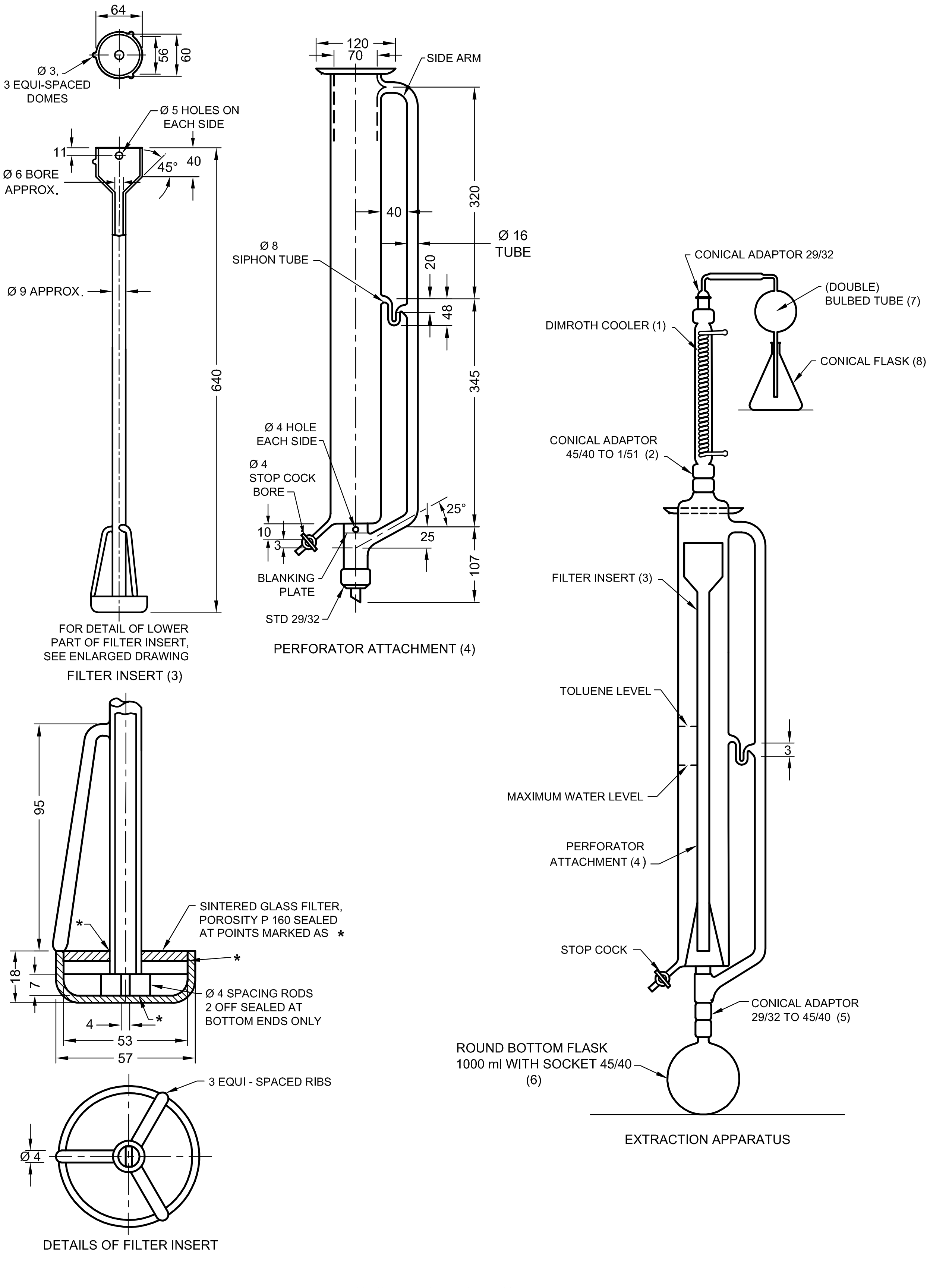
4.3 Water Bath, capable of maintaining a temperature of 40 ± 1 °C

4.4 Spectrophotometer, capable of measuring absorbance at 412 nm. A cell of 50 mm path length is recommended, especially for low emitting samples.

4.5 Extraction Apparatus

The apparatus shall consist of following (*see* Fig. 1):

a) Spiral condenser, total length approximately 400 mm; cone 45/40, socket 29/32 (Part 1).
b) Conical adaptor, socket 45/40, cone 71/51 (Part 2).
c) Filter insert, porosity P 160 (100 to 160 μ m), bowl and filter diameter 60 mm (Part 3).



All dimensions in millimetres.

Fig. 1 Extraction Apparatusfor Determinationof Formaldehyde Content in Partical Boards

d) Perforator attachment 1000 ml with cock (4 mm bore, socket 71/51, male 29/32) (Part 4).

e) Conical adaptor, socket 29/32, cone 45/40 (Part 5).

f) 1 000 ml round-bottomed flask, socket 40/50 (Part 6).

g) (Double) Bulbed tube, cone 29/32 (length approximately 380 mm), external diameter approximately 10 mm, bulb diameter approximately 50 mm, distance between bulb and bottom end of tube approximately 200 mm (distance between the bulbs approximately 50 mm) (Part 7).

h) Absorption bulb (for example, conical flask 250 ml) (Part 8).

4.6 Laboratory Equipment

The laboratory equipment shall consist of the following:

a) Volumetric flasks, 100 ml, 1 000 ml and 2 000 ml.

b) Conical flask, 250 ml.

c) Precision burette, 50 ml.

d) Volumetric pipettes, 1 ml, 2 ml, 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml, 100 ml.

e) Pipette, 25 ml.

f) Volumetric cylinders, 250 ml.

g) Two measuring cylinders, each 25 ml.

h) Watch glass, with diameter of about 120 mm.

j) Stoppered flasks, each 50 ml

k) Desiccator.

5 SAMPLING AND PREPARATION OF PIECES

5.1 Sampling

The test pieces are to be taken evenly distributed over the width of the (cooled) board excluding a strip of 500 mm width at a front side of the board. Take 18 test pieces of 25 mm X 25 mm X thickness of the board for the determination of the moisture content and a sufficient number of test pieces of the same dimensions to obtain approximately 500 g of the board for the extraction by perforator.

5.2 For Production Control

If this method is used for production control, the board selected for sampling is immediately cut up. The test pieces taken from the board shall be stored in a hermetically sealed container at ambient temperature. The formaldehyde determination should be carried out not more than 24 h after sampling.

5.3 For Other Purposes

If this method is used for other purposes, for example for board a already installed, the methods chosen for sampling, preparation of test pieces, and conditioning which influence the final result shall be as agreed between the parties and laid down in the test report. Unless otherwise agreed, the boards shall be conditioned for 43 days and the test pieces for 6 days at 27 ± 2 °C temperature and 65 ± 5 percent relative humidity.

6 PROCEDURE FOR EXTRACTION

6.1 Number of Determination

The determinations are to be carried out in duplicate.

The individual values of a duplicate determination may deviate by the maximum of 0.5 mg/100 g, but by not more than 10 percent related to the greater of the two single values; otherwise a third determination is to be carried out.

NOTE — For internal routine control, a single determination may be sufficient.

6.2 Determination of Moisture Content

The moisture content is determined on a sample of 5 to 6 test pieces (25 mm x 25 mm). The sample is weighed M_1, to an accuracy of 0.1 percent on a watch glass (about 120 mm diameter) and it is dried in a well-ventilated drying oven at a temperature of 103 ± 2 °C to constant mass, M_0 (about 12 h). Constant mass is regarded as having been reached when the mass of the test piece does not differ by more than 0.1 percent over a period of 4 h. After the removal from the drying oven, the test pieces shall be allowed to cool in a desiccator before weighing. The moisture content, M of the particle board, in percent, is calculated by the equation:

$$M = \frac{M_1 - M_2}{M_0} \times 100$$

6.3 Extraction in the Perforator

Before the apparatus is used the side arm of the perforator attachment shall be provided with thermal insulation in order to achieve circulation of toluene.

About 100 g of test pieces are weighed to an accuracy of 0.1 g, *m*, and put into the round bottom flask (*see* **4.5**). 600 ml of toluene (*see* **3.1**) is added. Subsequently, the round-bottom flask is connected to the perforator. About 1 000 ml of distilled water is poured into the perforator attachment, leaving a space of 10 to 20 mm between the surface of the water and the siphon outlet. Then the condenser and the gas absorption equipment are connected. The absorption bulb of the gas equipment (*see* **4.5**) is filled with about 100 ml distilled water.

When the apparatus is complete, the cooling and heating are turned on. Toluene must flow back regularly throughout the whole period of perforation, with a reflux rate of 50 to 70 drops per minute. Care must be taken that no water flows back from the absorption bulb (Part 8) into other parts of the equipment during the extraction process.

The extraction is carried on for 2 h, counting from the moment that the first bubbles pass through the filter insert. The heating shall be such that the onset of bubbling occurs between 20 and 30 min after turning on the heating device.

After these 2 h the heating is switched off and the gas absorption bulb is removed.

The water contained in the perforate is transferred, after cooling to ambient temperature, into the volumetric flask by means of the stopcock. The perforator is rinsed twice, each time with 200 ml of distilled water. The rinsing water is poured into the volumetric flask and the toluene discarded. The water contained in the absorption bulb of the gas absorption on equipment is poured into the flask. The volume of the water contained in the flask is then made up to 2 000 ml with distilled water to make up the formaldehyde solution.

6.4 Blank Test

The test shall be repeated without test pieces with new toluene from the same bottle as used for the perforation.

7 DETERMINATION OF FORMALDEHYDE CONTENT IN THE EXTRACT BY THE TITRATION METHOD

7.1 Procedure

Transfer 100 ml of the aqueous formaldehyde solution, prepared in **6.3**, in the volumetric flask into the conical flask. Add 50 ml of iodine solution (*see* **3.2**) and 20 ml of sodium hydroxide solution (*see* **3.5**). Close the flask and keep it in the dark for 15 min. Carefully add 10 ml sulphuric acid (*see* **3.3**). The liberation of excess iodine causes a brown colour to appear. Back titrate this excess iodine with the thiosulphate in the burette in the presence of starch (*see* **3.6**) as indicator

The presence of other products capable of being oxidized by iodine (ethyl alcohol, acetone, etc) shall be avoided.

7.2 Expression of Results

The formaldehyde content (perforator value), expressed in mg formaldehyde per 100 g of oven dry board, is calculated according to the following equation:

Formaldehyde content =

$$\frac{3.0\,(V_0 - V_1)\,(100 + M)}{m}\; mg/100\text{g}$$

where

V_0 = consumption of 0.010 mol/l thiosulphate solution for the blank test, in ml;

V_1 = consumption in ml of 0.010 mol/l thiosulphate solution for the test, in ml;

M = moisture content of the board, in percent; and

m = mass in g of test pieces before the extraction, in g.

NOTE — 1 ml 0.01 mol/l thiosulphate solution corresponds to 0.5 ml 0.01 mol/l iodine solution and 0.15 mg formaldehyde.

Results shall be rounded off to the next integer.

8 DETERMINATION OF FORMALDEHYDE CONTENT IN THE EXTRACT BY SPECTROPHOTOMETER METHOD

8.1 Procedure

From the aqueous formaldehyde solution (extract) prepared in **7.3**, 10 ml solution is taken with a pipette and added to 10 ml acetylacetone solution and 10 ml ammonium acetate solution in a 50 ml flask. The flask is stoppered, shaken and warmed for 15 min in a water bath at 40 °C. The greenish-yellow solution is cooled to room temperature, protected against the influence of light (for about 1 h). The absorbance of this solution is determined at a wavelength of 412 nm against distilled water using a spectrophotometer. A blank value is determined in parallel with distilled water and taken into consideration in the determination of the perforator value.

8.2 Calibration Curve

8.2.1 The calibration curve is produced from a standard formaldehyde solution, the concentration of which has been determined by iodometric titration (*see* **8.2.2**). The calibration curve shall be checked at least once a week.

8.2.2 *Standard Formaldehyde Solution*

Reagents used shall be as follow:

Standard iodine solution, concentration (l_2) = 0.05 mol/l

Standard sodium thiosulfate solution, concentration ($Na_2S_2O_3$) = 0.1 mol/l

Standard sodium hydroxide solution, concentration (NaOH) = 1 mol/l

Standard sulphuric acid solution, concentration (H_2SO_4) = 1 mol/l

Starch solution = 1 percent (*m/m)*

The solutions shall be standardized before use.

About 2.5 g formaldehyde solution (concentration 35 percent to 40 percent) is diluted in a 1 000 ml volumetric flask with distilled water and made up to the mark. The exact formaldehyde concentration is determined by iodometric titration as follows:

20 ml of the formaldehyde standard solution is mixed with 25 ml iodine solution and 10 ml sodium hydroxide solution. After 15 min standing, protected from light, 15 ml of sulphuric acid solution is added to the mixture.

The excess iodine is back-titrated with the thiosulfate solution. At the end of the titration, some drops of starch solution are added as indicator. The formaldehyde content is calculated by the following formula:

$$c\,(HCHO) = (V_0 - V) \times 15\,[c(Na_2S_2O_3)] \times 1\,000/20$$

where

$c(HCHO)$ = formaldehyde concentration, in mg/l;

V_0 = volume of thiosulphate solution for the blank test, in ml;

V = volume of thiosulphate titration solution for the test, in ml; and

$c(Na_2S_2O_3)$ = concentration of the thiosulphate solution, in mol/l.

NOTE — One millilitre (1 ml) 0.1 mol/l thiosulphate solution corresponds to 1 ml 0.05 mol/l iodine solution and 1.5 mg formaldehyde.

8.2.3 *Formaldehyde Calibration Solution*

Using the concentration determined in **8.2.2**, calculate the volume which will contain 15 mg formaldehyde. Transfer this volume, using a microburette, to a 1 000 ml volumetric flask and make up to the mark with distilled water. 1 ml of this calibration solution contains 15 mg formaldehyde.

8.2.4 *Determination of the Standard Curve*

Pipette either zero, 5, 10, 20, 50 or 100 ml of formaldehyde calibration solution into a 100 ml volumetric flask each and make up to the mark with distilled water. Ten millilitre (10 ml) of each dilution is analyzed photo-metrically by the same procedure as described in **8.1**. The absorbance values are plotted against the formaldehyde concentrations (between 0 and 0.015 mg/ml) on graph paper (*see* Fig. 2). The slope is either determined graphically or calculated.

8.2.5 *Expression of Results*

The formaldehyde content (perforator value), expressed in mg formaldehyde per 100 g of oven-dry board, is calculated by the following formula:

Formaldehyde content

$$= \frac{(A_S - A_b) \times f \times (100 + H) \times M}{m}\ mg/100g$$

where

A_s = absorbance of the analyzed extraction solution;

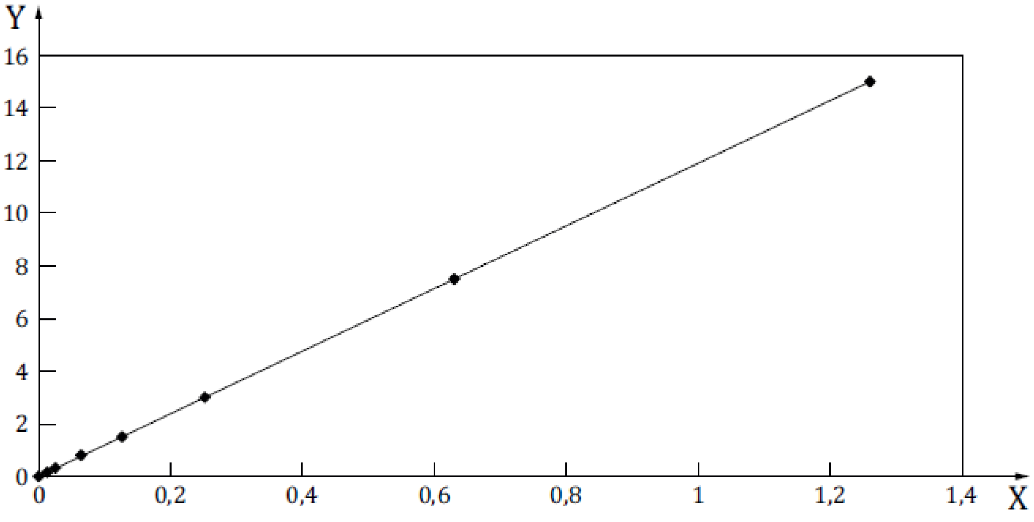
A_b = absorbance of an analysis with distilled or demineralized water;

f = slope of the standard curve, in mg/ml;

M = moisture content of the wood-based panel as a percentage;

m = mass in g of test pieces before the extraction, in g; and

V = volume of the volumetric flask (2 000 ml).



$c = f \times (A_S - A_b)$

a) Concentration of the diluted calibration solution (c)

b) Absorbance ($A_s - A_b$)

FIG. 2 EXAMPLE OF A CALIBRATION CURVE FOR FORMALDEHYDE DETERMINED BY ACETYLACETONE METHOD (PATH LENGTH 10 MM)

The formaldehyde content of a board is considered to be the mean value from the results of the two or three extractions. Results shall be expressed to one decimal place.

9 TEST REPORT

In the test report the following minimum information shall be given:

a) Origin of the board,
b) Place, location and state of the board at the time of sampling (in particular the moisture content),
c) Type of the board,
d) Thickness of the board (in mm),
e) Bulk density of the board (in kg/m^3),
f) Date of manufacture of the board,
g) Date of sampling,
h) Method of determination (by titration or spectrophotometer method),
j) Date of testing,
k) Moisture content (percent), at the time of testing, and
m) Perforator value (in mg formaldehyde/100 g dry board).

ANNEX A

(*Foreword*)

COMPOSITION COMMITTEE

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